

CHEMICAL ANALYSIS OF FLY ASH

1. SCOPE: This method is a modification of ASTM C-114, as referenced in ASTM C-311, for the chemical testing of fly ash by x-ray spectroscopy.
2. APPARATUS – MATERIALS:
 - 2.1. Philips MagiX PRO Wavelength Dispersive x-ray fluorescence spectrometer.
 - 2.2. SuperQ software
 - 2.3. Philips Perl'x 3 fused bead machine
 - 2.4. Platinum dish and crucible set
 - 2.5. Spex 8000 mixer/mill
 - 2.6. Lithium Bromide (LiBr): 10% solution non-wetting agent
 - 2.7. 67% Lithium Tetraborate (Li₂B₄O₇) : 33% Lithium Metaborate (LiBO₂) flux
3. PROCEDURE:
 - 3.1. Weigh approximately 2 grams of fly ash sample into a small beaker.
 - 3.2. Dry the sample to a constant weight in an oven at 110°C.
 - 3.3. Calculate the moisture content using the following formula:
$$MC = (A/B) \times 100$$
where: A = weight of sample after drying;
B = weight of original sample.
 - 3.4. Prepare porcelain crucibles by igniting at 750°C to a constant weight. Cool and store crucibles in a dessicator to avoid absorption of moisture
 - 3.5. Weigh 1.0 gram of fly ash sample from the moisture analysis accurately to 0.0001 grams into a prepared porcelain crucible. Ignite sample to a constant weight in a muffle furnace at 750°C and cool in a dessicator.

- 3.6. Calculate the loss on ignition (LOI) using the following formula:

$$\text{LOI} = (A/B) \times 100$$

where: A = weight of sample after ignition;
B = weight of moisture free sample.

- 3.7. Place at least 1 gram of fly ash sample from LOI in the spex 8000 mixer/mill and grind until a fine powder is obtained and/or sample is completely uniform. This takes approximately 30 seconds.
- 3.8. Weigh accurately to 0.0001 grams 6.0 grams of 67:33 flux directly into platinum crucible. Then weigh accurately to 0.0001 grams 0.6 grams of fly ash sample from the spex 8000 mixer/mill directly into the platinum crucible. Add 3 drops of LiBr solution. Place the platinum crucible and dish in the Perl'x 3 machine and select to run program 7* for all types of fly ash. This takes approximately 15 minutes. Program run is dependent on sample type.
- 3.9. Enter names of samples and LOI information in measure sample screen on the measure and analyze program.
- 3.10. Place sample in a 27mm steel cup. Then place in x-ray instrument and prepare to run fly ash application on the measure sample screen. Click on measure at the bottom of the screen. This may take a few minutes. The application chosen is dependent upon the sample type.
4. QUANTIFICATION: Program quantifies data by using a least squares program. Similar samples with known chemical makeups are used as standards in the quantification technique. As many standards as possible are used for best quantification. The results are reported as oxides in weight percents.
5. REPORT:
- 5.1. % Moisture Content
- 5.2. % LOI
- 5.3. % SO₃
- 5.4. % Al₂O₃
- 5.5. % Fe₂O₃
- 5.6. % R value: for class C fly ashes only. [(CaO – 5)/ Fe₂O₃]
- 5.7. % MgO
- 5.8. % Na₂O

5.9. % K₂O

5.10. % CaO

5.11. % SiO₂

* NOTE: Program 7 for fly ash includes: One oxidation for 4 minutes, temperature 1100°C, generator power 77, agitation angle 30, and agitation speed 15. One fusion for 5 minutes, temperature 1100°C, generator power 77, agitation angle 60, and agitation speed 20. Then a pause before casting for 10 seconds at a temperature of 1100°C. Casting time 10 seconds, temperature 1100°C, casting angle 123, casting speed 10, and time for solidification 2 minutes. Lastly there is natural air cooling for 4 minutes and forced air cooling for 2 minutes at a flow rate of 40. The setting of the dish height dial is 12/40 this depends on the size of the platinum dish being used.

APPROVED _____
Director
DIVISION OF MATERIALS

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